IS 7645 (1994): Phenyl J-acid, Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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भारतीय मानक

(Reaffirmed 2002)

फिनाइल जे-अम्ल, तकनीकी — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

PHENYL J-ACID, TECHNICAL — SPECIFICATION

(First Revision)

UDC 667·21 : 547·555

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

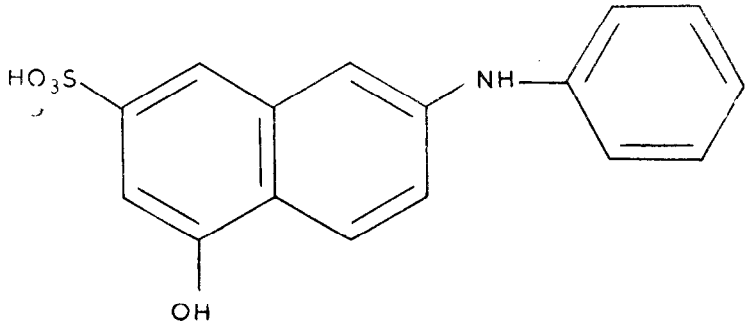
February 1994

Price Group 2

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Phenyl J-acid, chemically described as 2-phenylamino-5-naphthol-7-sulphonic acid ($C_{16}H_{13}O_4NS$) is an important intermediate used in the manufacture of dyes. It is also known as 2-phenylamino-5-hydroxy naphthalene-7-sulphonic acid. It is represented by the following structural formula:



PHENYL J-ACID
(MOLECULAR MASS 315)
CAS Registry Number [119-40-4]

This standard was first published in 1975. The Committee responsible for its preparation decided to update the standard in light of experience gained. In this version, the requirements of matter insoluble in dilute sodium carbonate and J-acid content have been modified.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 Rules for rounding off numerical values (*revised*). The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

PHENYL J-ACID, TECHNICAL — SPECIFICATION

(First Revision)

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for phenyl J-acid, technical.

2 NORMATIVE REFERENCES

The following Indian Standards contain provisions which through reference in the text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision and parties to agreement, based on the standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
797 : 1982	Common salt for chemical industries (*third revision*)
1070 : 1992	Reagent grade water (*third revision*)
2552 : 1989	Steel drums (galvanized and ungalvanized) (*third revision*)
5299 : 1969	Methods for sampling and testing for dye-intermediates

3 REQUIREMENTS

3.1 Description

The material shall be in the form of a paste, or, if dry, in the form of light grey to greenish grey powder.

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552 : 1989) lined with suitable polyethylene film, or as agreed to between the purchaser and the supplier.

4.2 Marking

Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;

Table 1 Requirements for Phenyl J-Acid, Technical

(*Clauses* 3.2, 5.3.1, 5.3.2 *and* 6.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Assay, percent by mass (on dry basis), *Min*	85	A
ii)	Aniline content, percent by mass (on dry basis), *Max*	1·0	B
iii)	J-acid content, percent by mass (on dry basis), *Max*	1·0	C
iv)	Matter insoluble in dilute sodium carbonate solution, percent by mass, *Max*	0·3	10.2 of IS 5299 : 1969

b) Indication of the source of manufacture;

c) Net mass of material;

d) Month and year of the manufacture;

e) Batch or lot number; and

f) The minimum cautionary notice worded as under:

"IT IS A MILD SENSITIZER. LOCAL CONTACT MAY CAUSE DERMATITIS"

4.2.1 The containers may also be marked with the Standard Mark.

4.2.2 The use of the Standard Mark is governed by the provisions of Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in 3 of IS 5299 : 1969.

5.2 Number of Tests

5.2.1 Tests for assay shall be conducted on each of the individual samples separately.

5.2.2 Tests for the determination of the remaining characteristics, namely, aniline content, J-acid content and matter insoluble in dilute sodium carbonate solution shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirement of assay if each of the individual test results satisfies the relevant requirement given in Table 1.

5.3.2 *For Composite Sample*

The lot shall be declared as conforming to the requirements of aniline content, J-acid content and matter insoluble in dilute sodium carbonate solution if the test results satisfy the relevant requirements given in Table 1.

6 TEST METHODS

6.1 Tests shall be conducted according to methods prescribed in Annexes and IS Specifications as given in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, 'pure chemicals' and distilled water (*see* IS 1070 : 1960) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Item* (i)]

DETERMINATION OF ASSAY

A-0 OUTLINE OF THE METHOD

A-0.1 The material is dissolved in dilute sodium carbonate solution. A known volume of the solution is coupled with standard 4-chloro-2-anisidine diazo in sodium carbonate and dilute sodium acetate medium and from the consumption of diazo, the strength is determined.

A-1 PREPARED SAMPLE

A-1.1 Dry the material at 105 ± 1°C to constant mass. Grind and mix well. Transfer the material to a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this prepared sample for tests.

A-2 REAGENTS

A-2.1 Alakaline H-Acid Indicator Solution

A-2.2 Brilliant Yellow Indicator Papers

A-2.3 Phenolphthalein Indicator Papers

A-2.4 Red RC Diazo Solution — 0·1 N.

A-2.5 Sodium Acetate Solution — 20 percent (*m/v*).

A-2.6 Sodium Carbonate Solution — 10 percent (*m/v*).

A-2.7 Common Salt — *See* IS 797 : 1967.

A-3 PROCEDURE

Weigh accurately about 15 g of the prepared sample (*see* **A-1**) and transfer to a 500 ml beaker with the help of water. Add slowly sodium carbonate solution to get a distinct red test on brilliant yellow paper and a faint pink test on phenolphthalein paper. Heat if necessary to get a clear solution. Transfer the solution to a 500-ml volumetric flask and make up the volume to the mark.

Pipette 50 ml of the solution into a 1-litre beaker. Add 20 ml of sodium carbonate solution and 100 ml of sodium acetate solution. Stir mechanically and cool with ice to 0 to 5°C. Titrate with standard red RC diazo solution from a cold water-jacketted burette. Add 15 g of common salt near the end point. Test with alkaline H-acid for excess diazo and with diazo for the coupling component. A faint test line with alkaline H-acid reproducible for a period of 5 minutes denotes the end point.

A-4 CALCULATION

$$\text{Assay, percent by mass, on dry basis} = \frac{V}{M} \times 31{\cdot}5$$

where

V = volume in ml of the standard red RC diazo solution used, and

M = mass in g of the prepared sample taken for the test.

ANNEX B

[*Table* 1, *Item* (ii)]

DETERMINATION OF ANILINE CONTENT

B-0 OUTLINE OF THE METHOD

Aniline is distilled and titrated with bromide-bromate solution.

B-1 REAGENTS

B-1.1 Bromide Bromate Solution — 0·1 N.

B-1.2 Hydrochloric Acid — 30 percent (*m/v*).

B-1.3 Potassium Iodide Starch Indicator Papers

B-1.4 Sodium Hydroxide Solution — 10 percent (*m/v*).

B-2 PROCEDURE

Dissolve 10 g of the prepared sample (*see* **A-1**) in 30 ml of sodium hydroxide solution and 400 ml of water. Transfer the solution to a 2-litre distillation flask and add 20 ml more of sodium hydroxide solution. Distil the aniline in the sample and collect the distillate in a series of receivers each containing 50 ml of water and 10 ml of hydrochloric acid. In each receiver, collect about 75 ml of the distillate and titrate with bromide-bromate solution adding 15 ml more of hydrochloric acid and using potassium iodide starch indicator paper. A titre value of about 0·2 ml of bromide-bromate solution indicates that the aniline content in the sample is practically distilled off. Sum up all the titre readings to know the total volume of bromide-bromate solution required for aniline.

B-3 CALCULATION

$$\text{Aniline content, percent by mass, on dry basis} = \frac{V \times N \times 1{\cdot}55}{M}$$

where

V = volume in ml of bromide-bromate solution used,

N = normality of bromide-bromate solution, and

M = mass in g of the prepared sample taken for the test.

ANNEX C

[*Table* 1, *Item* (iii)]

DETERMINATION OF J-ACID CONTENT

C-0 OUTLINE OF THE METHOD

J-acid is determined by using descending paper chromatographic technique.

C-1 APPARATUS

C-1.1 Chromatographic Sprayer

C-1.2 Developing Chamber

C-1.3 Micropipette

C-2 REAGENTS

C-2.1 Ammonium Hydroxide Solution — 1 percent (*m/v*).

C-2.2 Developer — *n*-propanol mixed with 20 parts by volume of water.

C-2.3 Phenyl J-Acid — free from J-acid.

C-2.4 J-Acid — pure.

C-2.5 Spray Reagent — 0·01 N solution of sulphanilic acid diazo diluted with 40 percent sodium acetate solution (*m/v*) in equal parts at the time of spraying.

C-3 PROCEDURE

C-3.1 First, prepare a standard solution of phenyl J-acid containing known amounts of J-acid. Into each of three 100 ml volumetric flasks, weigh accurately 1·0 g of phenyl J-acid. Then add 3·0 ml, 4·0 ml and 5·0 ml of 0·1 percent solution of J-acid in ammonium hydroxide solution to flask No. 1, 2 and 3 respectively. Dissolve the contents of the flask in ammonium hydroxide solution. Then make the volume up to the mark with ammonium hydroxide solution. Now, there are 3 solutions of 0·3, 0·4 and 0·5 percent J-acid content. In the fourth flask, weigh about 1·0 g of the prepared sample (*see* **A-1**), dissolve the ammonium hydroxide

solution and dilute to 100 ml with ammonium hydroxide solution.

C-3.2 Place 10 microlitre spot of each of the four solutions using micropipettes in the same line to a distance of about 4 cm on filter paper (Whatman No. 1 or equivalent). Place the paper in a descending paper chromatographic glass jar containing the developing reagent and previously saturated with the same developer. Allow the solvent to run in a descending manner for about 30 cm from the spot. This will take about 12 hours. Take out the paper after 30 cm run and dry the solvent completely. Then spray the paper with spray reagent. After 10 minutes of spray, compare the intensity of the J-acid spots visually with those of known standards.

C-4 CALCULATION

C-4.1 Report J-acid content as that which is nearest in intensity to the standard. In case, the colour intensity does not come in the range of standard spots, repeat the whole procedure using different percentages of J-acid.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act, 1986* to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such a review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards Monthly Addition'.

This Indian Standard has been developed from Doc: No. PCD 11 (1149)

Amendments Issued Since Publication

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